FRITTING OF REFRACTORY MATERIALS

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ABSTRACT. A high-temperature tensile testing machine with accurate recording of the stress-strain diagram, to determine the mechanical resistance of sintered refractory carbides of Groups IV and V transition metals, is described. Standard specimens of 30% residual porosity, prepared by sintering finely divided powders of tantalum, titanium, or zirconium carbide, were used. The tensile strength vs. temperature curve was located below the polycrystalline graphite curve for titanium and zirconium carbide, while it was above this curve for tantalum carbide, at all temperatures from room temperature to 3200°C. The high sensitivity to thermal shock of tantalum carbide is overcome by infiltration of porous skeletons with copper or silver, imparting resistance to thermal fluxes of 2000 w/cm² to the material and making it suitable for high-temperature applications.

The carbides of transition metals of Groups IV and V of the Periodic Table are characterized by extremely high melting points. This characteristic, very valuable for their use at high temperatures, requires sintering for preparing this type of material. The study of the sintering process is done by retractometry. In all cases, a blocking of the contraction is produced before the theoretical density is reached. The cause of this phenomenon is a coarsening of the grain.

The residual porosity is a function of the density before sintering. Besides this, minor additions may exert a considerable influence in the sense of an activation or, conversely, of an inhibition.

Sintering under load yields a better approach to the theoretical density. However, the process of macrocrystallization intervenes here and interrupts the shrinkage.

Numerous properties of refractory carbides have already been studied and published. However, literature data on the mechanical resistance at high temperatures are rare, fragmentary, frequently contradictory, and difficult to compare because of the discrepancy in the experimental conditions.

We invented and developed equipment for tensile testing at extreme temperatures, ensuring an accurate recording of the stress-strain diagram.

Specimens of standardized type, having a residual porosity of 30%, were pre-

 $[^]st$ Numbers in the margin indicate pagination in the foreign text.

pared by sintering fine powders of tantalum carbide, titanium carbide, or zir-conium carbide.

By means of these specimens, it became possible to plot the tensile strength curve as a function of the temperature for each of the three refractory components. For TiC and ZrC, these curves are distinctly below that of polycrystalline graphite which is the classical material still preferable in all cases unless it might be outranked in the future.

Conversely, for tantalum carbide, this curve is above the graphite curve over the entire temperature range, from room temperature to 3200°C.

This advantage would point toward interesting practical applications for this carbide, except that this material has a high sensitivity to thermal shock which, in this respect, makes it resemble sintered alumina.

Infiltration of porous skeletons by copper or silver permits removing this frequently eliminative drawback. Under these conditions, initial thermal fluxes as high as 2000 w/cm² can be tolerated. In studies of the solubility of tantalum carbide in copper and silver, it was found that the metal of infiltration (infiltrant) does not damage the refractory skeleton during its use.

A beneficial influence of the metal on the oxidizability of carbon is demonstrated in thermogravimetric tests.

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